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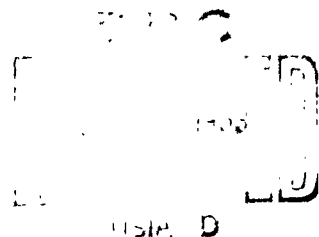
**FILM THICKNESS CONTROL
BY MEANS OF A
CRYSTAL RESONATOR**

Translation

(Work Assignment 34, Task 5a)

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FILM THICKNESS CONTROL BY MEANS OF CRYSTAL RESONATOR

This translation was prepared in response to AID Work Assignment No. 34, Task 5a. The article was originally published as follows:

Akishin, A. I., and V. S. Zazulin. Kontrol' tolshchiny plenok, poluchayemykh v vakuume, kvartsevym rezonatorom. Pribury i tekhnika eksperimenta, no. 1, 1963, 152-154.

The article describes a method of continuous control, by means of a quartz resonator, of the thickness of thin metallic films prepared in a vacuum by evaporation or any other means. The maximum sensitivity of the method is $\sim 10^{-9}$ g/cm². Calibration data are given for a 16-Mc quartz crystal. The method may also be applied to the study of cathode and certain surface phenomena.

A continuous control of film thickness and rate of film formation is frequently necessary in the process of manufacturing thin metallic and dielectric films in a vacuum by evaporation or by any other method. Such control is being accomplished in various ways (Ref. 1). Some of these, such as the method of weighing in a vacuum with a microbalance, are characterized by relatively low sensitivity (10^{-6} to 10^{-7} g) and are complicated in operation. The radioactive-isotope method has a very high sensitivity but a limited range of application. The present paper describes a sensitive method applicable to metallic films and based on the use of a high-frequency quartz resonator (Ref. 2,3). The natural frequency of the piezoelectric effect of a quartz plate depends upon the mass of the metallic electrodes deposited on the surface of the plate as well as upon its geometric dimensions and type of cut. The method of measuring the thickness of thin films deposited on the electrode surface of the quartz resonator is based on the measurement of the natural frequency shift caused by a change in electrode thickness. The technology of manufacturing quartz resonators has generally been based upon a method of accurate adjustment of the quartz plate frequency by varying the thickness of the metallic electrodes deposited on its surface (Ref. 4). The frequency of a quartz resonator, f , operating in the thickness dimension, d , of a quartz plate, is related to the geometric dimensions of the plate by:

$$f = N/d, \quad (1)$$

where N is the frequency factor determined by the type of cut and by the shape of the quartz plate. The increment of this function caused by a change in thickness is

$$\Delta f = - \frac{N \Delta d}{d^2} = - \frac{N \Delta m}{K \rho_q A d^2}, \quad (2)$$

where $\Delta d = \Delta m / K \rho_q A$, Δm is the increment of mass as a result of change in thickness of the metallic electrodes, ρ_q is the specific gravity of quartz, A is the surface area of the electrode serving as the substrate of the sputtered film, ρ_m is the specific gravity of the sputtered material, and $K = \frac{\rho_m}{\rho_q}$.

Substituting the value of d from equation (1), we obtain:

$$\Delta f = \Delta m f^2 / K N \rho_q A. \quad (3)$$

Since $f \gg \Delta f$, one can consider that f remains constant during the sputtering of films whose thickness is within the range 5000-10,000 Å. Hence,

$$\Delta f = B \Delta m, \quad (4)$$

where

$$B = \frac{f^2}{K \rho_q N A} \sim \text{const.}$$

Figure 1 shows the schematic diagram of the device. It is a bridge-type circuit and consists of a quartz-stabilized measuring oscillator, a reference oscillator, a mixer, and a frequency meter. The measuring-oscillator frequency is determined by the natural frequency of the quartz resonator used in the control of film thickness. The mixer separates the differential frequency, which is then measured by the frequency meter. The basic diagram of the measuring oscillator and mixer (Fig. 2) does not require explanation. The sensitivity and accuracy of the method can be determined by consideration of Equation (4) above and analysis of the capabilities of the individual instruments which constitute the device.

The sensitivity of the device is determined basically by the frequency stability of the measuring and reference oscillators. As is known, the stability of a quartz resonator is affected by changes in temperature. In the present case, the oscillator stability was $\sim 10^{-6}$. The thermal frequency coefficient of quartz resonators depends upon the cut of the quartz plate and its geometric dimensions.

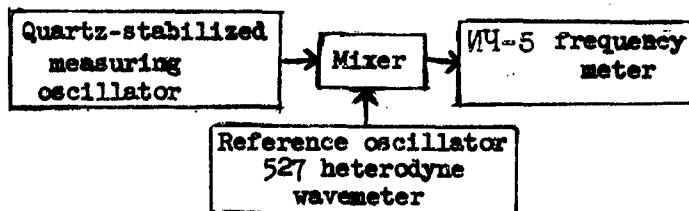


Fig. 1

The use of thermostats in the oscillator may increase the stability of the measuring circuits by an order of magnitude or more. In the selection of a quartz plate of a given cut, it is necessary to consider its frequency characteristics as well as the low value of its thermal frequency coefficient. The presence of parasitic resonances near the operating frequency of the measuring oscillator may lead to a spontaneous frequency shift to adjacent frequencies during the sputtering process and, consequently, may introduce an error. In this respect, the best frequency characteristics are found in quartz resonators having the lowest thermal frequency coefficient.

The stability of the reference oscillator has the same effect on the sensitivity of the device. In the present work the reference oscillator used was a 527 heterodyne wavemeter with a stability of 2 cps/°C with respect to the quartz heterodyne and ± 20 cps with respect to a heterodyne with continuous frequency control over a short interval of time (10 to 15 min). The differential frequency was measured by an M4-5 frequency meter.

The above relationship shows that the instruments used in this work afford the possibility of achieving a sensitivity of $\sim 10^{-9}$ g/cm² for an operating frequency $f \sim 20$ Mc when measuring the thickness of thin metallic films obtained in a vacuum. The quartz resonators used to determine the thickness of sputtered films were calibrated in an YBP sputtering machine. The quartz plates of the resonators had a cut of AT 36°20'. The sputtered material was deposited simultaneously on the metallic electrode of the quartz resonator through a 4-mm diaphragm and on a mica control plate 20 x 10 mm. The control plate and the quartz resonator electrode were placed

at approximately the same distance from the source of the sputtered material. The mica plate was carefully weighed on an analytic microbalance before sputtering. The reference oscillator was used to bring the measuring bridge into balance before the beginning of the sputtering operation. The oscillator frequency decreased in the course of sputtering the metallic films on the electrode of the quartz resonator. The material was allowed to sputter on the quartz resonator electrode until the bridge was unbalanced at $\Delta f = 30-50$ kc. After sputtering, the mica plate was weighed again to determine the weight of the sputtered material per unit of plate surface; this made it possible to find the mass of the film deposited on the surface of the metallic electrode of the quartz resonator.

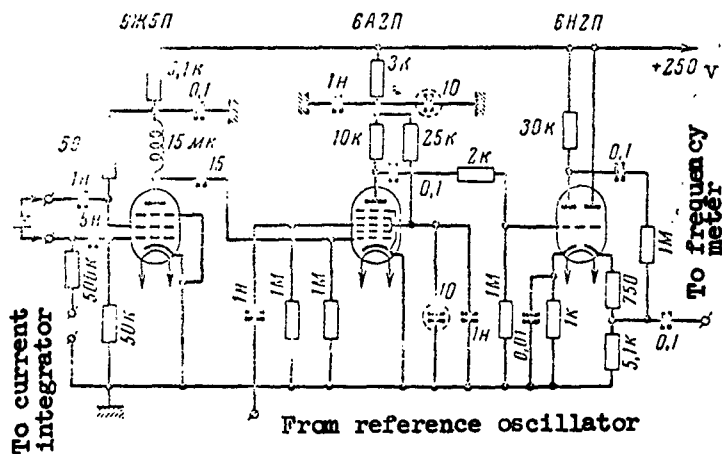


Fig. 2.

About 20 independent calibrating runs were made with quartz resonators with a fundamental frequency $f \approx 16$ Mc. The mean sensitivity of a quartz resonator is

$$\Delta m / \Delta f \approx 2 \cdot 10^{-7} \text{ g/kc} = B^{-1}, \quad (5)$$

The electrodes of quartz resonators were sputtered with Al, Ag, and Cu; here, within the limit of accuracy of our measurements,

$$\Delta m / \Delta f \approx \text{const}$$

that is, the sensitivity of a quartz resonator does not depend upon the nature of the sputtered material. Equations (4) and

(5) can thus be written in the following form for a quartz resonator with a natural frequency $f \approx 16$ Mc:

$$\Delta m = \Delta f/B = 2 \cdot 10^{-7} \Delta f \text{ g/kc.}$$

The electronic equipment used made it possible to determine a frequency shift of ± 20 cps. Consequently, quartz resonators can be used to measure an electrode mass increment $\sim 10^{-9}$ g/cm².

The maximum deviation of the fundamental frequency of the quartz resonator in the course of sputtering metal on resonator electrodes should not exceed several hundred kilocycles, as the operation of the resonator may become unstable or cease altogether if sputtering is continued. The use of low-frequency quartz resonators decreases the sensitivity of the method but permits the measurement of thicker films. Repeated use of quartz resonators required the removal of the sputtered material by chemical means or by cleaning the electrode with fine abrasive powder.

The quartz resonator was used to measure the rate of carbon film formation on its electrode by means of an electron beam with a density of 1 $\mu\text{a}/\text{cm}^2$ and an energy of 500 ev. The quantity of electricity accumulated on the electrode was measured by an electron current integrator (Fig. 2). The electron beam was applied in a dismountable metal vacuum vessel with rubber seals. The vessel was evacuated by means of a IJB-100 oil diffusion pump to a pressure of 10^{-5} mm Hg without using a nitrogen trap. Under these conditions, the rate of formation of carbon film on the exposed surface was $\sim 2 \cdot 10^{-7}$ g/mm² $\mu\text{a} \cdot \text{hr}$. It follows from these data that the electron incident upon the electrode surface interacts on the average with several molecules of oil or other organic materials adsorbed on the surface.

Quartz resonators may also be used in the study of cathode sputtering, etc. (Ref. 2).

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